

# PATENT SPECIFICATION

NO DRAWINGS

L150.408



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Date of Application and filing Complete Specification: 7 Sept., 1967.

No. 40977/67.

Application made in Switzerland (No. 13026) on 8 Sept., 1966.

Complete Specification Published: 30 April, 1969.

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Index at acceptance: —C2 C(1K1C3, 1K1E); D1 P(1C1A, 1C4, 1D, A1, B2B1, B5A, B5B, C2A12A5, C2C2, C2C3, C2C8, C2C9, C2C11, C2C17)

Int. Cl.:—C 07 d 55/04

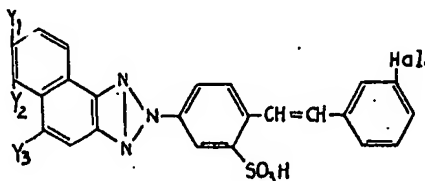
## COMPLETE SPECIFICATION

### Stilblyl-Naphthotriazoles and Their Use as Optical Brightening Agents for Textiles

#### ERRATUM

SPECIFICATION NO. 1,150,408

Page 6, line 73, for existing formula read



THE PATENT OFFICE  
1 October 1971

R 1

25 adie. In contrast thereto, the modern resin finishing of textiles to attain stability of form which is known and popular particularly in America by the terms Wash-and-Wear and No-Iron, presents problems which are more difficult to solve and, to some extent, are still unsolved. This is particularly the case for the finishing of textiles by the so-called permanent press process which is now becoming popular. One of these processes consists in impregnating the cotton fabric to be processed with a mixture of resinous precondensates, preferably of an acid amide-formal resin basis, particularly of the type carbonic acid diamide-formal resin, a curing catalyst and also softeners and then drying under mild conditions. The fabric finished in this way then goes to the manufac-

65 Pfersee, Augsburg (West German Republic); "FIXAPRET CPN", Badischen Anilin- und Soda-fabrik, (BASF), Ludwigshafen (West German Republic), or "PERMAFRESH Reactant 183", Warwick Chemical Corporation, Wood River Junction, (USA). Other resins used in practice are alkyl carbamates, dimethylol triazones, 1,3-diglycidyl glycerol, and also imidazolone/triazine mixed condensates. The activity of the curing catalyst plays an important part in the permanent press process. Only highly active catalysts can be used; zinc nitrate is preferred to zinc chloride or magnesium chloride, which latter two compounds, in general, are not completely satisfactory.

80 Unfortunately it has been found that zinc nitrate causes by far the greatest yellowing

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575 576 577 578 579 580 581 582 583 584 585 586 587 588 589 590 591 592 593 594 595 596 597 598 599 600

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Int. Cl.:—C 07 d 55/04

## COMPLETE SPECIFICATION

### Stilbyl-Naphthotriazoles and Their Use as Optical Brightening Agents for Textiles

We, J.R. GEIGY A.-G., a body corporate organised according to the laws of Switzerland of 215 Schwarzwaldallee Basle, Switzerland, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention concerns textiles and cellulose fibres finished and optically brightened by the permanent press process, processes for the production of these articles, optical brighteners suitable for this process and processes for the production thereof.

The optical brightening of cellulose fibres in any stage of processing, whether it be in the form of yarn, fabrics or articles, before or after the usual finishing of such textiles, as well as in use on washing or rinsing after washing, does not pose any basic technical problems even though improvements, e.g. with regard to light fastness, shade and brilliancy of the brightener, are certainly desirable.

In contrast thereto, the modern resin finishing of textiles to attain stability of form which is known and popular particularly in America by the terms Wash-and-Wear and No-Iron, presents problems which are more difficult to solve and, to some extent, are still unsolved. This is particularly the case for the finishing of textiles by the so-called permanent press process which is now becoming popular. One of these processes consists in impregnating the cotton fabric to be processed with a mixture of resinous precondensates, preferably of an acid amide-formal resin basis, particularly of the type carbonic acid diamide-formal resin, a curing catalyst and also softeners and then drying under mild conditions. The fabric finished in this way then goes to the manufac-

turer, is cut and made up into clothing. The garment is then pressed at a temperature of about 120°C and then the resin is condensed and cured by a heat treatment for about 15 minutes at 160—180°C. Compared with the previous resin finishing of textiles of the wash-and-wear type, the permanent press finishing, which has become known particularly under the protected trade name KORATRON, owned by Koret, San Francisco, USA, is distinguished by substantially improved retention of shape after repeated washing. There is also the so-called pre-curing method of performing the permanent press process in which cross linkage is done before making up and the garment is pressed after making up.

Resins suitable for the permanent press finishing are particularly compounds from the group of dimethylol-N,N'-alkylene ureas, for example commercial products which are available under the protected trade names such as "KNITTEX everfit LE", Chemische Fabrik Pfersee, Augsburg (West German Republic); "FIXAPRET CPN", Badischen Anilin- und Soda-fabrik, (BASF), Ludwigshafen (West German Republic), or "PERMAFRESH Reactant 183", Warwick Chemical Corporation, Wood River Junction, (USA). Other resins used in practice are alkyl carbamates, dimethylol triazones, 1,3-diglycidyl glycerol, and also imidazolone/triazine mixed condensates. The activity of the curing catalyst plays an important part in the permanent press process. Only highly active catalysts can be used; zinc nitrate is preferred to zinc chloride or magnesium chloride, which latter two compounds, in general, are not completely satisfactory.

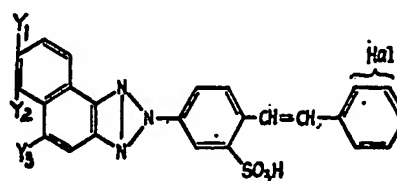
Unfortunately it has been found that zinc nitrate causes by far the greatest yellowing

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of the textile material of all usual catalysts under the necessary working conditions. Thus, in the permanent press finishing of white goods the use of optical brighteners to maintain a pleasant appearance of the goods is unavoidable because the yellowing can neither be washed out nor removed by chemical bleaching. In addition, it has also been found that the preferred optical brighteners for cellulose fibres from the class of 4,4'-bis-(triazinylamino) - stilbene - 2,2' - disulphonic acids, which contain high performance compounds with regard to affinity to the fibre, brilliancy and shade of the fluorescence, cannot be used because, under the reaction conditions for the resin curing, zinc nitrate so alters the optical brighteners that, on subsequent exposure, the goods become strongly brown coloured. The white goods treated are unusable because the browning cannot be removed either by one or repeated washing or by oxidative or reductive chemical bleaching. Actually, it is possible to reduce the browning on exposure to light by the choice of another curing catalyst; but then, however, the important advantage of the new finishing process is lost, i.e. the very good retention of shape of the finish goods on frequently repeated washing. Many other known optical brighteners cannot be used either because of lack of sufficient affinity to cellulose, insufficient improvement of the degree of whiteness, causing of undesirable tinges or because of insufficient stability under the working conditions. Also the optical brightening of the resins used leads to unsatisfactory results and, in addition, demands an undesirable after-treatment of the stabilised goods. In particular, their treatment with the active optical brighteners of the 4,4' - bis - (triazinylamino) - stilbene-2,2'-disulphonic acid series has no effect due to lack of affinity to the resin-treated fibre. For this reason, permanent press finishing is limited in practice to previously dyed fibres. But it is just for this very reason that, in the case of white goods such as blouses and shirts, there is a great need for a finishing which imparts permanence of shape and is fast to washing.

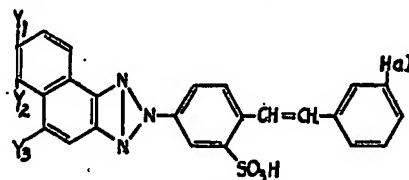
Surprisingly, in accordance with the present invention it has now been found that textiles can be finished by the permanent press process and simultaneously optically brightened if, before or during the impregnation with an aqueous preparation containing curable resins or resin precondensates, resin catalyst and, optionally, softener and subsequent drying, stabilising and curing, a stilbyl-naphthotriazole compound of the general formula I, or a water soluble alkali metal or ammonium salt thereof,



is drawn onto the goods. In this formula: one of the symbols  $Y_1$ ,  $Y_2$  and  $Y_3$  represents the sulphonyl acid group and the other two represent hydrogen, and "Hal" represents F, Cl or Br.

The halogen substituent, i.e. fluorine, bromine or chlorine, can be in the o- or m-position to the vinylene bridge; it has a particularly favourable influence in the m-position.

Specially preferred optical brighteners correspond, therefore, to the general formula II

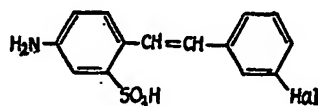


wherein the symbols has the meanings given in formula I.

The second sulphonyl acid group is advantageously in the position of  $Y_2$ . In particularly valuable optical brighteners according to the invention the two characteristics last mentioned are both present and, finally, chlorine is the preferred halogen substituent "Hal".

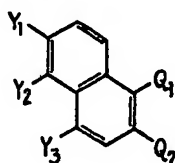
Chiefly the alkali metal salts are used as soluble salts of optical brighteners of the general formula I or II; however, also ammonium salts, particularly N-substituted ammonium salts of strong nitrogen bases can also be used, e.g. alkanolamine salts.

The new, particularly valuable stilbyl-naphthotriazole compounds of general formula II are obtained by known methods, e.g. by coupling a diazo compound of a 4-aminostilbene compound of the general formula III



with 1- or 2-aminonaphthalene monosul-

phonic acid of the general formula IV where-  
by coupling occurs in the o-position to a  
primary amino group Q<sub>1</sub> or Q<sub>2</sub> in the com-  
pound of the general formula IV



(IV)

In the formulae III and IV, "Hal", and Y<sub>1</sub>,  
Y<sub>2</sub>, and Y<sub>3</sub> have the meanings, including the  
preferences, given above. One of Q<sub>1</sub> and Q<sub>2</sub>  
represents the amino group and the other  
represents hydrogen which can be replaced  
under the coupling conditions of the stilbyl  
azo group. The o-aminoazo dyestuffs so ob-  
tained are then oxidised by known methods  
to the corresponding stilbyl triazole com-  
pounds of general formula II, for example  
with oxygen or gaseous mixtures containing  
oxygen such as air, in the presence of copper  
tetramine salts, or with soluble salts of  
hypochlorous acid such as sodium, potassium  
or calcium hypochlorite.

In the form of their water-soluble alkali  
metal salts, the new stilbyl-naphthotriazole  
compounds of formula II are pale yellow  
coloured powders which dissolve well in water  
and have good affinity to cellulose under the  
usual dyeing conditions. They serve chiefly  
for the optical brightening of cellulose fibres  
in aqueous liquors and, in particular, can also  
be used in the impregnation liquors for the  
permanent form finishing of cellulose fabric  
by the permanent press process.

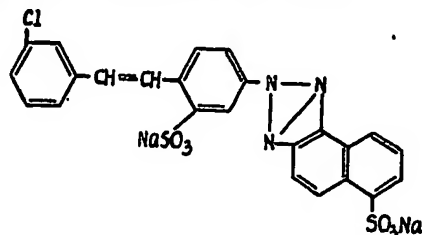
The optical brighteners of formula I used  
according to the invention are drawn onto the  
fibre in the finishing of cellulose fabric by  
the permanent press process in usual amounts  
of 0.01 to 0.3% of active substance, calculated  
on the weight of the fibres. This is done, for  
example, by mixing them in amounts of 0.1  
to 3.0 g per litre in impregnation liquors  
which contain 100—250 g of N,N' - di-  
methylol - ethylene - dihydroxy urea and  
5—20 g of zinc nitrate per litre, padding the  
goods in a foulard with a wet pickup of about  
70—100%, calculated on the dry weight of  
the fabric, then drying at 60—80°, making  
up into garments, pressing and curing.

Further details can be seen from the fol-  
lowing examples which illustrate the subject  
of the invention. Where not otherwise stated,  
parts are parts by weight. The temperatures  
are given in degrees Centigrade.

## EXAMPLE 1

A cotton fabric is impregnated in a foulard  
with a suspension containing 180 g of a di-  
methylol - dihydroxy - ethylene urea (KNIT-

TEX everfit IE, Chem. Fabrik, Pforsee, Augs-  
burg, Germany), 10 g of zinc nitrate and 1.0  
g of the brightener of the formula



in 1000 ml of 25° warm water, squeezed out,  
(wet pickup about 70%) and dried at 60°. 60  
The fabric so treated is then treated for 15  
minutes at 160° to attain condensation and  
cross-linkage of the resin. In this way a fabric  
is obtained which, on the one hand has per- 65  
manence of form and, on the other, has a  
brilliant white shading. Such optically  
brightened fabric can be exposed to light  
without danger of browning.

A weaker brightening effect is obtained if, 70  
instead of 1.0 g, only 0.5 g of the optical  
brightener is used.

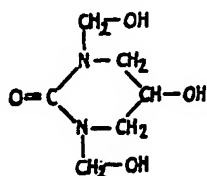
If in the above example, the cotton fabric  
is replaced by staple fibre fabric then the 75  
fabric so treated is also strongly optically  
brightened, it has permanence of form and  
does not go brown on exposure to light. Also,  
if a mixed fabric consisting of cotton and  
polyester or cotton and synthetic polyamide  
fibres is used then the cellulose part is also 80  
strongly optically brightened. The polyester  
or polyamide part is brightened in a separate  
pre-treatment using a brightener which is  
specifically suitable for these fibres.

A similar optical brightening is obtained 85  
if, in this example, the 0.5 g of disodium  
salt of 2 - (3''' - chlorostilbyl - 4'') -  
naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5' -  
disulphonic acid is replaced by equal parts of  
the disodium salt of 2 - (3''' - chlorostilbyl - 90  
4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole -  
2'',4' - or 2'',6' - disulphonic acid or (2 -  
(3''' - fluorostilbyl - 4'') - (naphtho - 1',2':  
4,5) - 1,2,3 - triazole - 2'',5' - disulphonic 95  
acid.

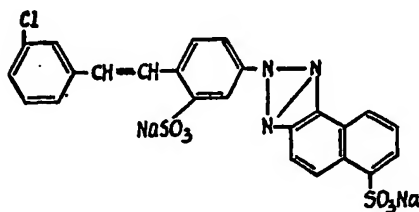
In addition, if 0.5 g of the disodium salt  
of 2 - (2''' - chlorostilbyl - 4'') - (naphtho -  
1',2':4,5) - 1,2,3 - triazole - 2'',5' - disul-  
phonic acid or of the disodium salt of 2-(2'''-  
chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 100  
1,2,3 - triazole - 2'',4' - or 2'',6' - disul-  
phonic acid is used as optical brightener,  
then somewhat less brilliant white shadings  
are obtained.

## EXAMPLE 2

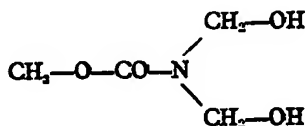
A cotton fabric is impregnated in a foulard  
with a suspension containing 150 g of di-  
methylol-oxypropylene urea of the formula



12 g of zinc nitrate and 1.0 g of the optical brightener of the formula



- 9 in 1000 ml of 25° warm water, squeezed out, (wet pickup about 70%), and dried at 60°. To attain condensation and cross-linkage of the resin, the fabric so treated is treated for 15 minutes at 165—168°. In this way a fabric is obtained which, on the one hand has permanent form and, on the other, has a brilliant white shading. A fabric optically brightened in this way can be exposed to light without danger of browning.
- 10 A similar but weaker white shading is obtained if, in the above example instead of the disodium salt of 2 - (3''' - chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5' - disulphonic acid, 0.5 g of the disodium salt of 2 - (2''' - chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5' - disulphonic acid is used as optical brightener
- 25 In addition, in the above example, the dimethylol-oxypropylene urea can be replaced by 200 g of dimethylol-methylcarbamate of the formula

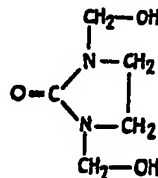


- 30 An optically brightened fabric of permanent form which does not go brown in light is also obtained.

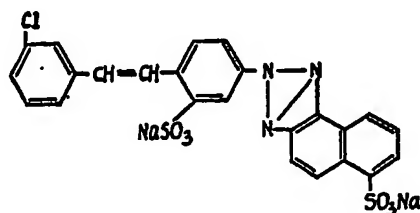
#### EXAMPLE 3

A cotton fabric is impregnated in a foulard with a suspension containing 200 g of a mix-

ture of 70 parts of dimethylol-ethylene urea of the formula



and 30 parts of hexamethylol melamine, 8 g of zinc nitrate and 1.0 g of the optical brightener of the formula



in 1000 ml of 25° warm water, squeezed out, (wet pickup about 70%), and dried at 60°. The fabric so treated is then treated for 15 minutes at 165—170° to attain condensation and cross-linkage. In this way a fabric is obtained which, on the one hand, has permanent form and, on the other, has a brilliant white shade. Fabric so optically brightened can be exposed to light without danger of browning.

A similar but weaker white shading is obtained if, in the above example, instead of the disodium salt of 2-(3'''-chlorostilbyl-4'')-(naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5'-disulphonic acid, 0.5 g of the sodium salt of 2 - (2''' - chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5'-disulphonic acid are used as optical brightener.

In addition, in the above example, the mixture of dimethylol-ethylene urea and hexamethylol melamine can be replaced by 190 g of dimethylol-propylene urea. A form-stabilised, optically brightened fabric showing no browning in light is also obtained.

#### EXAMPLE 4

A mixed fabric consisting of 65% polyethylene glycol terephthalate and 35% cotton is impregnated at 25° in a foulard with a suspension which contains 190 g of a dimethylol-dihydroxy-ethylene urea (KNIT-TEX everfit LE, Chem. Fabrik Pforsee, Augsburg, Germany), 10 g of zinc nitrate, 1.0 g of the optical brightener of the formula

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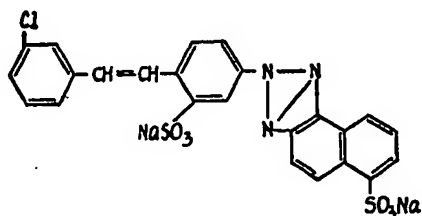
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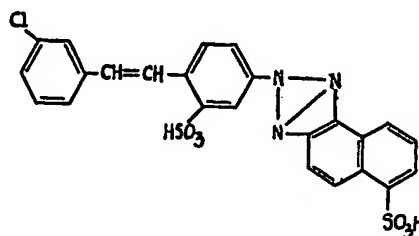
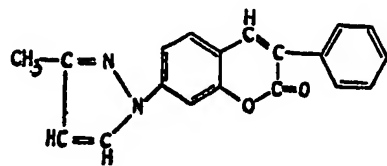
stable, optically brightened fabric is obtained which shows no browning in light. 45

If, instead of the zinc nitrate mentioned in the above example as curing catalyst, equal amounts of zinc chloride or magnesium chloride are used and otherwise the procedure described above is followed, then optically brightened fabrics are also obtained. Fabrics treated in this way have less permanence of form but in light they also shown no browning effects. 50

#### EXAMPLE 5

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and 0.6 g of the optical brightener of the formula



5 in 1000 ml of water, squeezed out, (wet pick-up about 65%), and dried at 90—92°. To polymerise, the fabric is then exposed to a temperature of 140—150° for 5 minutes. If desired, the fabric so treated can now be made up into garments. 10

For the final form stabilisation, the fabric is first steamed for 15—20 seconds and then pressed for 15—20 seconds at 205—215° and a pressure of about 800 g/sq. cm to break open the cross linkage present and to prevent new cross linkage formation. 15

In this way a fabric is obtained which, on the one hand has permanent form and, on the other, has a white shading. Fabric optionally brightened in this way can be exposed to light without danger of browning. 20

A weaker brightening effect is obtained if, in the above example, instead of 1.0 g, only 0.5 g of the optical brightener are used and otherwise the same procedure is followed. In addition, a similar white shading is obtained if, instead of 0.5 g of the disodium salt of 2'(3''' - chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',5' - disulphonic acid used as optical brightener, 0.5 g of the disodium salt of 2-(3'''-chlorostilbyl-4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',4'- or -2'',6'- disulphonic acid, or the disodium salt of 2'(2''' - chlorostilbyl - 4'') - (naphtho - 1',2':4,5) - 1,2,3 - triazole - 2'',4'- or -2'',5'- or -2'',6'- disulphonic acid are used. 25 30 35

Further, in the above example, the dimethylol-dihydroxy-ethylene urea can be replaced by 180 g of a mixture consisting of 70 parts of dimethylol-ethylene urea and 30 g of hexamethylol melamine or by 200 g of dimethylol methyl carbamate. Here too, a form- 40

A solution of 31.0 g of the sodium salt of 4 - amino - 3' - chlorostilbene - 2 - sulphonic acid and 7.1 g of sodium nitrite in 800 ml of water is diazotised indirectly at a temperature of 8—10° by pouring it into a solution of 12.8 g of hydrochloric acid in 120 ml of water. The whole is then stirred for 30 minutes at 8—10° whereupon the excess nitrite is removed with a small quantity of sulphamic acid and the diazo suspension is added at a temperature of 12—15° to a solution of 22.4 g of 2-aminonaphthalene-5-sulphonic acid, and 4.1 g of sodium hydroxide in 600 ml of water. The pH of the coupling is kept at about 4.5 by the addition of crystallised sodium acetate and the temperature is kept at 20—25°. On completion of the coupling, the o-aminoazo dyestuff is completely salted out by the addition of 140 g of sodium chloride and washed with 10% sodium chloride solution. The moist dyestuff is then dissolved in 1000 ml of hot water with the addition of sodium hydroxide solution until the reaction is clearly phenolphthalein alkaline and then a solution of 2.5 g of crystallised copper sulphate in 100 ml of water and 10 g of monoethanolamine are added. The o-aminoazo dyestuff is then oxidised by the introduction of a strong air current at a temperature of 92—95° while stirring vigorously to form the stilbylnaphthotriazole compound. After the red colour of the o-aminoazo dyestuff has disappeared, excess aqueous sodium sulphide is added to the solution and precipitated copper sulphide is removed by filtration. The copper residue is washed with 250 ml of hot water and the combined filtrates are heated to 95°. To remove any colouring products, 5—10 g of sodium hydrosulphite are added until the 55 60 65 70 75 80 85 90 95

solution remains clear and then 5% of sodium chloride is added. After stirring for 4 hours at room temperature, the precipitated product is filtered off and washed with 5% sodium chloride solution. If necessary, the compound can be further purified by recrystallisation from 50% aqueous n-propanol or by dissolving and again precipitating from water. The product obtained is dried in vacuo at 120—125°.

The disodium salt of 2-(3'''-chlorostilbyl-4'')-(naphtho-1',2':4,5)-1,2,3-triazole-2'',5'-disulphonic acid is obtained as a yellowish powder which dissolves well in water and melts at over 300°.

The compound obtained is a valuable brightening agent for the optical brightening of cellulose fibres, synthetic polyamide fibres, soap, soap powders and detergents. The compound mentioned is especially suitable for the optical brightening of cellulose fibres which, subsequently or simultaneously, are to be impregnated with a resin using zinc nitrate as catalysts, dried, possibly partially condensed and, after being made up into garments, again condensed and cross-linked at temperatures of 160—180°.

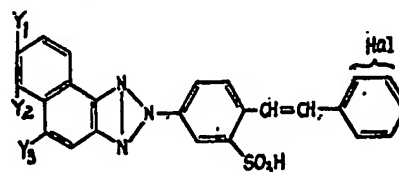
Compounds having similar properties, some of which have a somewhat different shade, are obtained if in the above example, the 2-aminonaphthalene-5-sulphonic acid is replaced by equal amounts of 2-aminonaphthalene-6- or -7-sulphonic acid or 1-aminonaphthalene-4-sulphonic acid. In this case, the disodium salt of 2-(3'''-chlorostilbyl-4'')-(naphtho-1',2':4,5)-1,2,3-triazole-2'',6'- or -2'',7'- or -2'',4'-disulphonic acid respectively is obtained as a yellowish to pale yellowish powder which melts at over 300°.

The words FIXAPRET and PERMA-FRESH used in this specification are Registered Trade Marks.

#### WHAT WE CLAIM IS:—

1. Process for the finishing and optical brightening of textiles by the permanent press process by impregnation with aqueous preparations containing curable resins or resin precondensates and curing catalyst, drying, stabilising and curing, characterised by drawing on to the goods to be finished, before or during the impregnation, a stilbyl naphthotriazole compound of the general formula I,

or a water-soluble alkali metal or ammonium salt thereof,



wherein one of the symbols  $Y_1$ ,  $Y_2$  and  $Y_3$  represents the sulphonyl acid group and the two others represent hydrogen and

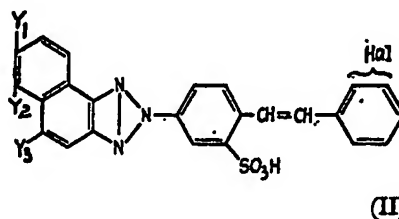
"Hal" represents chlorine, fluorine or bromine and is in the o or m position to the vinylene group.

2. Process according to claim 1 characterised in that "Hal" is in the meta-position to the vinylene bridge.

3. Process according to claim 2, characterised in that "Hal" represents chlorine.

4. Process according to claim 1 characterised in that  $Y_2$  represents the sulphonyl acid group and  $Y_1$  and  $Y_3$  represent hydrogen.

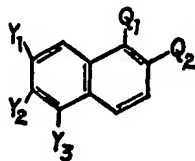
5. Stilbyl naphthotriazole compounds of formula II



wherein one of  $Y_1$ ,  $Y_2$  and  $Y_3$  represents the sulphonyl acid group and the others represent hydrogen, and "Hal" represents fluorine, chlorine, or bromine.

6. Compounds according to claim 5 wherein "Hal" represents chlorine.

7. Process for the production of stilbyl naphthotriazole compounds of formula II characterised by coupling the diazo compound of a 4-amino-3'-halo-stilbene-2-sulphonyl acid in which the halogen is F, Cl or Br, with an aminonaphthalene monosulphonyl acid of the general formula IV whereby coupling occurs in the o-position to the amino group  $Q_1$  or  $Q_2$  in the compound of the general formula IV.



## (IV)

- 5 which couples in the o-position to a primary amino group  $Q_1$  or  $Q_2$  wherein one of  $Y_1$ ,  $Y_2$ , and  $Y_3$  represents the sulphonic acid group and the remaining symbols represent hydrogen, to form the o-aminoazo dyestuff and oxidising this by methods known *per se* to form the naphthotriazole compound of formula II.
- 10 8. Process for the finishing and optical brightening textiles by the permanent press process as claimed in claim 1 substantially as

described with reference to any of the foregoing examples 1 to 4.

9. Brightened textiles substantially as described with reference to any of the foregoing examples 1 to 4. 15

10. Manufacture of stilbyl naphthotriazole compounds of formula II as defined in claim 5 substantially as described with reference to the foregoing example 5. 20

11. Stilbyl naphthotriazole compounds of formula II as defined in claim 5 whenever prepared or produced by the processes of manufacture particularly described. 25

12. Stilbyl naphthotriazole compounds of formula II as defined in claim 5 as identified in the foregoing example 5.

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